



The Synthesis of Poly (Ethylene Oxide) Macromonomers

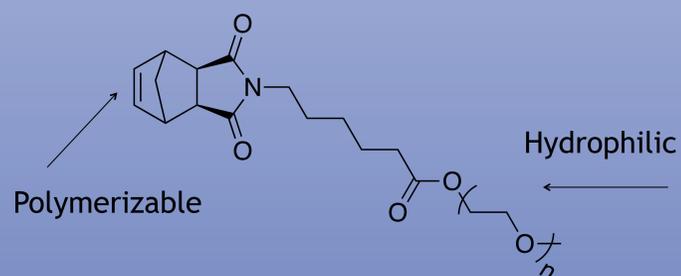
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Introduction:

The development of nanotechnology is essential for the improvement of current devices used throughout many important fields including medicine, electronics and energy. While the capabilities to advance these areas vary, it is evident that in order to improve current materials there is a need to precisely control structure morphology in 2 and 3 dimensions on nanometer-length scales. The importance of being able to control the size, shape and dimensions of nanomaterials opens the door to new research in the creation of dynamic systems that can assemble into designated materials. The research conducted was the initial steps toward the attainment of precise nanomaterials through the development of an efficient monomer synthesis for future use in polymeric materials. Hydrophilic/hydrophobic interactions can be used to control hierarchical assemblies of polymers to obtain precise nanostructures. This involved the synthesis of a monomer with (poly-ethylene) oxide introducing a hydrophilic element to this monomer. The macromonomer produced can be combined with commercially available hydrophobic monomers for synthesis of polymers with various architectures.



Experimental Design:

A multistep synthesis was performed starting with 5-Norbornene-2,3-dicarboxylic anhydride to yield norbornene carboxylic acid imide. The reaction of the imide with poly(ethylene glycol) methyl ether yielded the PEO Macromonomer. The starting material and the desired product are shown. Each product was analyzed with ¹H NMR to verify purity and reaction success. The goal of this experiment is to design an efficient and scalable route to the desired monomer shown above.

Results and Discussion:

In the experiment conducted the formation of the poly(ethylene oxide) macromonomer was successful. The changing of the stereochemistry of the endo-5-Norbornene-2,3-dicarboxylic anhydride to the *exo*-anhydride yielded about 50% of the desired product, according to proton NMR (Figure 1). This impure product was carried to the next step by reacting with 6-amino hexanoic acid. This reaction was high yielding with little unreacted starting material remaining (Figure 2). The final synthetic step involved the reaction of the norbornene carboxylic acid imide with poly(ethylene glycol) methyl ether yielding the desired product of a poly(ethylene oxide) containing monomer (Figure 3).

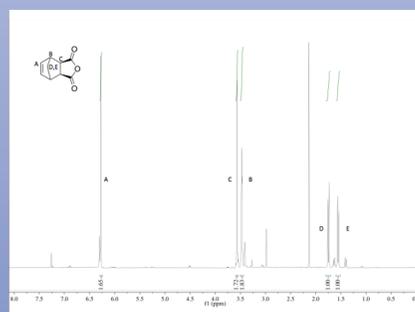
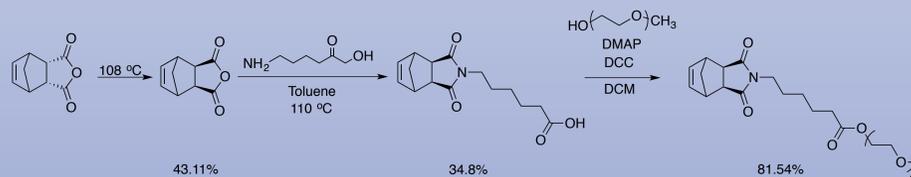


Figure 1. ¹H NMR Spectra of Purified Norbornene *exo*-anhydride

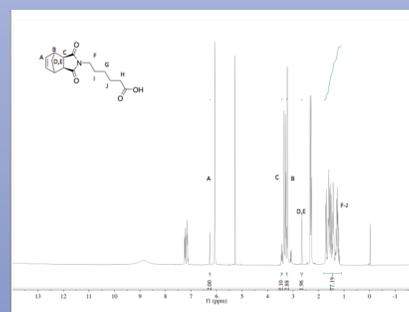


Figure 2. ¹H NMR Spectra of Purified norbornene carboxylic acid imide

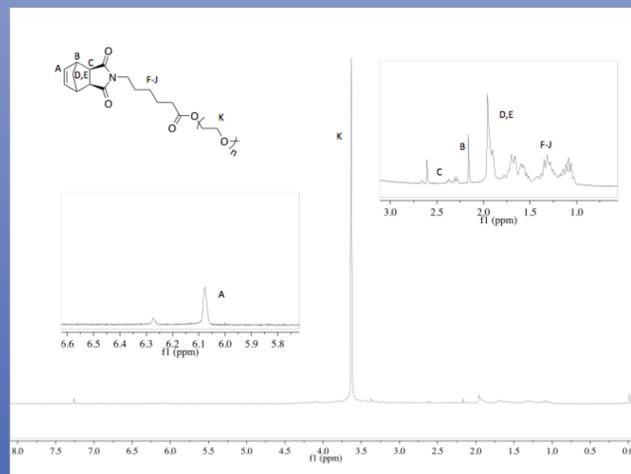
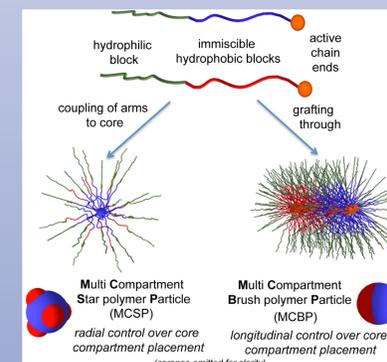


Figure 3. ¹H NMR Spectra of PEO Macromonomer

Future Work:

Once the desired poly(ethylene oxide) macromonomer is isolated it can be polymerized with various comonomers. Different polymer architectures can be explored to produce both star and brush polymers.¹ The precise synthesis of polymers to modify the surface of nanodevices for targeted applications has been one of the major focuses in the community for decades.² These polymer-based nanostructures could potentially self assemble, offering the attractive feature of assembly or disassembly on demand in selective solvents.³



Conclusions:

The synthesis of the poly(ethylene oxide) Macromonomer was completed, however. With the formation of the hydrophilic macromonomer the steps toward the formation of nanomaterials can be taken. More of the monomer will have to be made under similar conditions as to have a similar outcome of yields for the monomer produced.

Acknowledgements:

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References:

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